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One-Liter Test: A Mid-Scale Safety Characterization Test For Melt-Castable Explosives

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13. ABSTRACT (Maximum 200 words)

The One-Liter test was developed as a mid-scale safety characterization test specifically for use with melt-castable explosives. It is a one-dimensional test carried out under relatively unconfined conditions that are similar to those encountered in melt casting operations. The test allows the researcher to assess the response of the explosive to a controlled thermal stimulus under laboratory-like conditions and, more importantly, to determine its critical temperature. The latter is defined as the lowest constant surface temperature at which a material of a specific size, shape and composition can self-heat catastrophically. This test allows one to validate kinetic and thermal parameters determined by standard small-scale laboratory experiments. These parameters are necessary to predict the critical temperatures associated with large scale melt cast operations. The experimental critical temperature of the explosive used in this report to demonstrate the use of the One-Liter test falls in the temperature range 152-155 °C. Critical temperatures for large-scale melt casting operations up to 100 gallons are predicted.

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PREFACE

ACKNOWLEDGEMENTS

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

SECTION I

INTRODUCTION

1. BACKGROUND

All energetic materials decompose exothermically when exposed to elevated temperatures (above absolute zero) for prolonged times, such as during large-scale melt-casting operations. The rate of this chemical decomposition reaction is governed by the laws of chemical kinetics. If it decomposes according to a single first-order rate process, its internal temperature will be described by the following equation (Reference 1):

$$-\lambda \nabla^2 T + \rho C(\partial T/\partial t) = \rho Q Z e^{-E/RT}$$

where λ is thermal conductivity (cal K^{-1} cm^{-1} sec^{-1}), ∇^2 is the Laplacian operator, T is temperature (K), ρ is density (g cm^{-3}), C is heat capacity (cal g^{-1} K^{-1}), Q is heat of decomposition (cal g^{-1}), E (cal mol^{-1}) and Z (sec^{-1}) are the activation energy and pre-exponential, respectively, from the Arrhenius equation and R is the ideal gas constant (1.987 cal mol^{-1} K^{-1}).

When the decomposition process produces heat faster than it can be dissipated to the surroundings, the energetic material will begin to self-heat and this will lead to a decomposition event that may be violent (rapid combustion) or catastrophic (explosion). Safe formulation and processing operations, coupled with storage and usage requirements, require that the energetic materials/formulations response to heat stimulus be known. Frank-Kamenetskii (Reference 2) and Chambré (Reference 3) have solved the above equation under the steady state condition $\partial T/\partial t = 0$, obtaining the following expression for critical temperature in terms of the related physical parameters:

$$T_c = E/R\ln(a^2 \rho Q Z E/T_c^2 \lambda \delta R)$$

where T_c is the critical temperature, E and Z are the activation energy and pre-exponential from the Arrhenius equation, a is a dimension (e.g., radius of a sphere or infinite cylinder or half thickness of an infinite slab), ρ is the density, Q is the heat of decomposition (not detonation or combustion), R is the ideal gas constant, λ is the thermal conductivity and δ is a shape factor (3.32 for spheres, 2.0 for infinite cylinders, 0.88 for infinite slabs and 2.7-2.8 for cylinders with length equal to diameter). The critical temperature is defined as the lowest constant surface temperature at which a material of a specific size, shape and composition can self-heat catastrophically. This latter equation, usually referred to as the Frank-Kamenetskii (F-K) equation, is solved by iteration and usually provides a conservative (worst-case) prediction of the T_c for operations in a melt kettle (Reference 4).

It is important for the developer to realize that a more accurately predicted value can be gained for a particular process if the kinetic parameters used in the F-K equation correspond to the specific chemical reaction step that governs the process. Confidence in the accuracy of a predicted critical temperature can be gained by conducting the One-Liter test, provided the predicted value for the melt kettle size required to prepare the sample is sufficiently high to assure a safe operation. The One-Liter test was developed at the Energetic Materials Branch of the Munitions Directorate, Air Force Research Laboratory, Eglin AFB, FL, to be used exclusively as a pre-scale-up safety characterization test for melt-castable formulations. The One-Liter test essentially characterizes the entire self-heating process. This includes the temperatures associated with the initiation of non-catastrophic and of catastrophic (critical temperature) self-heating, the temperature at which the catastrophic event occurs and insight into the severity of the thermal decomposition reaction under conditions that are similar to those associated with a melt-casting operation.

2. OBJECTIVE

The objective of this report is to describe the One-Liter test and the data analysis technique. The latter will be accomplished by analyzing the raw data acquired from an actual test (Reference 5) with fine grain octol, a mixture containing 65 and 35 percent by weight HMX (classes I and II) and TNT, respectively.

SECTION II

EXPERIMENTAL PROCEDURES

1. TEST ARRANGEMENT:

a. Reaction Vessel

A predetermined amount of energetic material/formulation, hereinafter referred to as the sample, is cast directly into a one-liter, Pyrex, round-bottom flask. The sample weight used in this test was 1411.1 grams.

b. Temperature Measurement

A thermocouple bundle is pre-positioned in the molten sample along the centerline extending from the neck to the bottom of the flask and secured in place until the entire sample has solidified. The thermocouple bundle is constructed with five or six thermocouples (Type k) encased in stainless steel sleeves. They are of decreasing length with the longest touching the bottom of the flask. The tips of the remaining thermocouples are placed approximately 31.5, 64, 95.5 and 127.5 mm from the bottom of the flask. This design places the tips of the thermocouples at the bottom, center and top of the spherical portion of the flask and mid-way between the bottom /center and top/center locations. A thermocouple bundle positioned in a one-liter flask loaded with explosive (solid state) is depicted in Figure 1. If a sixth thermocouple is used, it is positioned in the neck of the flask 228.5 mm from the bottom. The bundle is secured in place during the test by a clamping device placed on top of the oven. This clamping device also provides shielding from the draft caused by the upper portion of a circulating fan that cools the fan motor positioned on the top of the oven. There is also a single thermocouple used to acquire the internal oven temperature. All thermocouples are calibrated by using an OMNI-CAL Temperature Calibrator, Model No. 8A-110, manufactured by Omega Engineering, Inc.

c. Heating Method

While the method used for heating the flask containing the sample is optional, this facility utilizes a disposable, plywood (5/8-inch thick) oven. It is equipped with a four-element electric resistive heater that is driven by an Omega Proportional Temperature Controller, Model 650. The four elements are mounted horizontally between the back wall of the oven and a baffle separating them from the main section containing the one-liter flask. The height of the baffle is 14 inches thereby allowing a one-inch wide gap at the bottom and top for air circulation through the main oven chamber. The elements are placed approximately 8 inches above the floor in a rectangular volume with width, height and depth dimensions of 16, 16 and 6 inches respectively. The inner blade assembly of a small, disposable, double-bladed fan circulates air through the main chamber of the oven by pushing the air through the heated elements. The front door of the oven consists of two

0.375-inch thick glass panes positioned 0.5-inch apart. The panes are held in place by grooves cut into the plywood. The glass front allows the reaction flask to be observed by video camera during the test. The oven is pre-conditioned prior to the test by holding the internal portion at approximately 110 °C for 24 hours. This procedure essentially degasses and slightly chars the internally exposed wood minimizing the clouding of the inside surface of the glass during the early part of the test. After heat conditioning, all seams in the oven are sealed with a heat-resistant, siliconized caulking material. The inside of the oven is lighted during the test by using an externally placed floodlight.

The top of the oven is sectioned parallel to the door and through the centerline of an opening specially designed to fit around the neck of the one-liter flask. This allows the removal of the front section of the top so the neck of the flask can be placed in the opening. This construction effectively secures the flask in place during the test. A picture of the experimental setup with the loaded one-liter flask positioned in the oven is shown in Figure 2.

d. Data Acquisition

Thermocouple data are collected in real-time (15-second intervals) by using a computerized data acquisition system. A canned program converts the acquired thermocouple voltage to temperature (°C).

2. PROCEDURE FOR THE ONE-LITER TEST

a. Sample Liquid Density Determination

The liquid density of the sample must be determined over a temperature range that includes the processing temperature and is of sufficient length to afford a density/temperature curve from which an extrapolation to the predicted critical temperature can be made. This experiment can be accomplished by any suitable method and may be as simple as heating a known sample mass in a graduated cylinder. The liquid density of the octol sample, obtained at 82.6 °C, was 1.67 g cm^{-3}.

b. Critical Temperature Prediction

The F-K equation, shown again below,

$$T_c = E/R\ln(a^2\rho QZE/T_c^2\lambda\delta R),$$

is used to provide critical temperature predictions for energetic materials/formulations of specific sizes and geometries. For convenience of the reader, the components of this equation for the one-liter geometry are defined again below:

T_c is the critical temperature (K),
E is the energy of activation for the thermal decomposition reaction (cal mol^{-1}),

R is the ideal gas constant (1.987 cal K^{-1} mol^{-1}),
a is the radius of the one liter sphere (6.123 cm),
ρ is the density of the energetic formulation (g cm^{-3}),
Q is the heat of the thermal decomposition reaction (cal g^{-1}),
Z is the pre-exponential factor for the thermal decomposition reaction (s^{-1}),
λ is the thermal conductivity of the energetic formulation (cal cm^{-1} s^{-1} K^{-1}) and
δ is the shape factor for spherical geometry (3.32).

The values for the above parameters can be obtained by direct measurement or acquired from the literature. The largest and smallest values normally appearing in the F-K equation are the Arrhenius activation energy and associated pre-exponential and thermal conductivity and are therefore the most critical to know accurately (Reference 6). While the activation energy and pre-exponential are difficult to determine for the specific chemical reaction step associated with catastrophic decomposition (critical temperature), a factor leading to the highest accuracy, they can be determined for global thermal decomposition by using the method described in ANSI/ASTM E698-79 (Reference 7). This method, hereinafter referred to as Method 1, usually leads to a conservative, but useful predicted critical temperature. In a test involving a mixture, it may be useful to use activation energy and pre-exponential values associated with the most reactive component obtained by an independent analytical technique (Reference 1). This second method is hereinafter referred to as Method 2. Both methods for acquiring these important input parameters for use in the F-K equation were used in this report.

A measured thermal conductivity for the energetic material/formulation being tested is preferred, but difficult to obtain for conditions replicating those of the test or processing operation. Such being the case, it has been shown to be acceptable to use an estimated value, generally one in the range 2e-4 to 13e-4 cal s^{-1} cm^{-1} K^{-1} (References 4 and 6). We find it beneficial to use the two extremes to establish a critical temperature range for a selected size and geometry or to establish a range of critical temperatures for increasing sizes for a given geometry. Critical temperatures were also predicted for the one-liter size by using literature thermal conductivity values of 7e-4 (Reference 6) and 12e-4 cal cm^{-1} sec^{-1} K^{-1} (Reference 7).

Since most explosives have heats of reaction for the decomposition process in the range, 300 to 750 cal g^{-1}, it has been shown to be acceptable to use 500 cal g^{-1}, unless this parameter can be directly measured or is available from the literature (References 4 and 6). For this report a value of 325 cal g^{-1} was used, which is simply the accepted value shown above adjusted by the weight percent of HMX in the composition (Reference 1).

c. Estimated Maximum Sample Volume

It is desirable that the volume of the sample at the critical temperature, which will be at least partially molten, be confined to the spherical portion of the one-liter flask or, at least, not spill from the flask. Sample spillage may come in contact with hot components associated with the oven, e.g., circulating fan and/or heating coils, and result in premature

ignition. The maximum volume of sample (V_{max}) to be cast into the one-liter reaction flask is estimated by the following procedure: 1) Determine the internal volume of the spherical portion of the one-liter flask (V_f) by filling that portion of the flask with a measured amount water and 2) multiply the ratio of liquid density of the sample at the processing temperature (ρ_{PT}) to the extrapolated density at the critical temperatures (ρ_{CT}) by the volume of the spherical portion of the one-liter flask.

$$V_{max} = V_f \, (\rho_{PT}/\rho_{CT})$$

Since the sample is introduced into the reaction flask in the molten state, a marker should be placed on the reaction flask to indicate the position of V_{max}. The upper level of molten sample should not exceed the level indicated by this marker.

d. Sample Preparation

As previously mentioned, the optimal method for loading the explosive into the one-liter flask is by direct casting of a predetermined amount of molten sample. This method sometimes causes a problem during the initial heating phase of the test in that the thermal expansion of the solid casting prior to the start of the remelting process may be greater than that of the glass, thereby breaking the flask before melting begins. An alternate method of loading is to introduce the sample in powder or chunk form directly into the flask. A potential problem associated with this latter method is that the bulk volume of the solid sample that equates to the estimated V_{max} usually exceeds that of the flask, including the neck. Also, it is difficult to insert the thermocouple bundle into the sample in solid form or to load the solid sample around a pre-inserted thermocouple bundle. The octol used in this test was cast into the flask.

e. Sample Conditioning

It is advantageous to have the sample in the molten state prior to the dynamic portion of the test to ensure that the change in the slope of the temperature/time curve associated with the melting process does not mask slope changes associated with the self-heating process. Therefore, as a standard procedure, the sample is heated at maximum rate (approximately 5 °C min^{-1}) to a predetermined temperature that is above the melting/processing temperature and then held until the sample (center thermocouple) and oven temperatures equilibrate (normally 5 to 7 hours). This hold temperature can be as high as 50 to 60 °C below that of the predicted critical temperature (T_c). By completion of this isothermal period the continuous phase (binder portion) of the sample should be in the molten state.

f. Heating Rate

At the completion of the isothermal period, the sample is heated at a rate of 3.3 °C hour^{-1} (Reference 8) until observable thermal decomposition occurs or the test is terminated for other reasons.

SECTION III

RESULTS AND DISCUSSION

a. Temperature/time Profiles

An example of temperature/time data acquired during the first 3000 seconds of this example One-Liter test carried out with octol (65/35) is shown in Figure 3. These temperature/time data can then be transformed into temperature/time traces, such as the two traces shown in Figure 4, which are from the thermocouples located in the oven and in the center of the molten octol sphere. These two traces cover the entire time of the test from initial temperature ramp to the catastrophic event. Examples of the information that can be gleaned from these traces (see Figures 5 through 8) are, (1) initiation of melting, (2) sample heat flow characteristics, (3) temperature associated with the initiation of non-catastrophic self-heating, (4) critical temperature and (5) temperature at which the catastrophic thermal decomposition event takes place.

b. Sample Conditioning

The octol sample, 1411.1 grams, was heated at a rate of approximately 5 °C min^{-1} to a measured average hold temperature of 101.9 °C (designated hold temperature was 101 °C), which is 63.8 °C below the predicted critical temperature (165.7 °C) reported in Reference 5. The temperature ramp rate was maintained until near the designated hold temperature, when the temperature controller engaged to minimize temperature overshoot. In this case, the overshoot was 12.5 °C and the time period above 101.9 °C was approximately 35 minutes. The sample was conditioned at this temperature for a period of 6.73 hours. The temperature/time traces associated with all of the thermocouples located in the sample for both the ramp and isothermal periods, along with ten data points associated with the oven thermocouple, are shown in Figure 5. The positions associated with the overshoot maximum temperature and with the initiation of the isothermal and the melting periods are indicated by markers and by associated slope changes. Since the self-heating process is believed to be most severe at the center location of an energetic material in a given configuration under slow heating conditions, the temperature/time trace associated with this thermocouple along with that of the oven are shown in Figure 6 for clarity. The first three sections of the sample trace, I, II and III, are associated with solid state sample heating during the initial temperature ramp (I), oven cooling from the temperature overshoot (II) and the isothermal period prior to the initiation of sample melting (III). Section (IV) is associated with melting (80-81 °C) of the TNT component (35 percent by weight) of the sample. The sample temperature is assumed to have equilibrated with the oven temperature in section V. The slope of section IV is greater than that expected for an ideal melting process, since the formulation contains 65 percent by weight HMX that is still in the solid state and continues to heat. The relative difference between the two thermocouple readings (101.9 and 91.1 °C) in section V is sample dependent. All thermocouples were calibrated prior to starting the test and are assumed to be correct. The temperature indicated at the

initiation of sample melting (83-84 °C) as compared to that acquired by DSC analysis (81.0 °C) supports this assumption.

c. Detection of Self-Heating

Following the conditioning period, the oven temperature is increased at a linear rate (R_o) of 3.3 °C hr^{-1}. The sample, after a short equilibration period, is assumed to heat at a linear rate (R_s) that is governed by heat-transfer characteristics specific to the sample. It is presumed that $R_s < R_o$ and, as a result, the sample temperature-time traces will diverge from that attributed to the oven. The shape of the sample temperature-time traces can also be affected by self-heating (exothermic chemical decomposition) and by cooling (vaporization) processes. The initiation of self-heating will cause the sample temperature-time trace to converge with that of the oven, while sample cooling will cause the traces to diverge.

Examples of temperature-time traces from thermocouples located in the oven and in the sample (bottom, center and top of the sphere), covering the last 60000 seconds of the programmed heating phase of the test, are shown in Figure 5. The vertical line passing through the three sample traces is positioned at an observable slope change that is attributed to the onset of the self-heating process, which is presumed to be non-catastrophic. This line separates the near-linear portion of the traces attributable primarily to the sample heat-transfer characteristics ($R_s < R_o$) from the more turbulent portion that is typical of bubbling that may occur during the exothermic chemical decomposition process.

The subtle changes in slope shown in Figure 7 can be greatly enhanced by subtracting the sample temperature from the oven temperature ($T_o - T_s$) for the entire dynamic period of the test and plotting this "delta temperature" (ΔT) versus time (Figure 8). As previously discussed, the temperature-time curves associated with the oven and the sample diverge if $R_s < R_o$. The ΔT-time curves will therefore have a positive slope. That the slope will be positive is more clearly envisioned by the relationship $\Delta T = t(R_o - R_o)$, where t is increasing. The two traces shown in Figure 8 were obtained by using the temperature-time data from the oven, sample center (blue) and sample bottom-center (red) thermocouples. These curves overlay one another and have been divided into four sections. Section I is associated with the heating differential discussed earlier and is represented by a linear trace with a positive slope. The increased slope of Section II is believed to be caused by volatilization of the TNT binder. Section II terminates at 142 °C due to the initiation of non-catastrophic self-heating. This process continues (Section III) until the onset of catastrophic self-heating in the 152-155 °C temperature range. Catastrophic self-heating, a self-perpetuating process, continues until a catastrophic exothermic decomposition event occurs.

d. Catastrophic Event

The dynamic portion of the test, following the conditioning period, ended abruptly at a catastrophic combustion event that occurred at a temperature of 167 °C (oven temperature of 184.2 °C). The entire test, that includes the initial heating, conditioning and dynamic periods, lasted for 33 hours. The entire sample was consumed during the combustion event.

e. Predicted Critical Temperatures

As discussed in Sub-section 2.b. of this Section, critical temperatures were predicted by using both Methods 1 and 2. All input parameters used to calculate critical temperatures for spherical geometry are shown in Table 1. Four thermal conductivity values were chosen to be used for these calculations, 2e-4, 13e-4, 12e-4 and 7e-4 cal cm^{-1} s^{-1} K^{-1}. The first two were used with both methods and are thusly shown in the "Common" column. The first of the latter two was used specifically with Method 1 and the final value specifically with Method 2.

Table 1. Parameters Used to Calculate Critical Temperatures for Spherical Geometry.

Parameter	Parameter Values		
	Method 1	Common	Method 2
E (cal mol^{-1})	50200[1]		52700[2]
Z (s^{-1})	1.49e19[1]		5e19[2]
a (cm)(radius)		0.032 to 76.0	
Q (cal g^{-1})		325[3]	
δ (spherical)		3.32	
R (cal mol^{-1} K^{-1})		1.987	
ρ (g cm^3)		1.67[4]	
λ (cal cm^{-1} s^{-1} K^{-1})	12e-4[5]	2e-4 and 13e-4[2]	7e-4[2]

(1) Reference 7 (values attributed to the HMX component).
(2) Reference 6 (literature values for pure HMX).
(3) Reference 1 (adjusted as 0.65(500).
(4) Reference 5 (determined at 82.6 °C).
(5) Reference 9 (literature value for octol 75/25).

Prior to the undertaking of large-scale melt-casting operations the critical temperatures for a series of spherical containers of increasing diameter are predicted. This size range includes the size(s) anticipated for the scaled up operations. These predictions, shown in Table 2, were accomplished by using the parameters shown in Table 1. The various critical temperatures that were predicted by using Methods 1 and 2, each with thermal conductivities of 2e-4 and 13e-4 cal cm^{-1} s^{-1} K^{-1}, are presented graphically in Figure 9 as best-fit power trendlines. Specific data points associated with thermal conductivity values of 12e-4 (Method 1) and 7e-4 (Method 2) cal cm^{-1} s^{-1} K^{-1} are also shown in Figure 9. As can be seen from the data presented in Table 2, the critical temperature predicted by using Method 1 with a thermal conductivity of 2e-4 cal cm^{-1} s^{-1} K^{-1} closely matches the experimental critical temperature range (152 vs 152-155 °C). Critical temperatures that are predicted for the 30- and 100-gallon melt kettles by using the data from the Method 1

column in Table 2 along with a thermal conductivity of 2e-4 cal cm^{-1} s^{-1} K^{-1} are 122 and 118 °C, respectively. Assuming a processing temperature of 85 °C, the safety factors associated with these large-scale melt-casting operations are 37 and 33 °C, respectively, and are considered to be only marginal.

Table 2. Calculated (F-K Equation) and Experimental Critical Temperatures (T_c) Versus Sample Diameter (Spherical Geometry) for Specific Thermal Conductivities.

Diameter (cm)	Critical Temperature (°C)						Experimental
	Calculated						
	Method 1			Method 2			
	2e-4	13e-4	12e-4	2e-4	13e-4	7e-4	
0.064	247.7	269.6		260.3	282.1		
1.0	192.7	210.1		205.3	222.6		
2.0	180.7	197.1		193.2	209.7		
4.0	169.2	184.8		181.7	197.4		
10.0	155.0	169.6		167.4	182.0		
12.246[1]	**152.0**	**166.3**	**165.7**	**164.3**	**178.8**	**173.9**	**152-155**
20.0	144.9	158.7		157.1	171.1		
30.0	139.2	152.6		151.3	164.9		
40.0	135.2	148.4		147.3	160.6		
108.0	122.1	134.4		134.1	146.6		
152.0	117.8	129.8		129.7	141.9		

(1) Inside diameter of the one-liter, round-bottom flask used for this test.

f. Thermal Decomposition Reaction Severity

The severity of the catastrophic event may vary from a simple combustion event to a more violent process, e.g., explosion or detonation. The response of octol (65/35) to this thermal insult was observed to be a mild combustion event where the entire sample was consumed, leaving the glass, one-liter flask deformed, but still in one piece.

g. Data Reported

The minimum data reported should include the specific experimental conditions, temperatures and times associated with all self-heating processes and the severity of the final decomposition event. A more detailed data package should contain a graphical representation of the temperature/time history and of the special thermogram from which self-heating can be extracted and the complete listing of the temperature/time values.

h. Time to Explosion Test

The Time to Explosion test (Reference 10) offers the researcher an opportunity to determine the critical temperature of an energetic material on a laboratory scale. This value for octol (65/35) is 236 °C (Reference 5). The critical temperature for octol (65/35) in one-liter geometry, as previously discussed, falls in the range 152-155 °C. The thermal conductivity range that corresponds to this latter temperature range is 1.97e-4 to 2.95e-4 cal cm^{-1} s^{-1} K^{-1}. By using this thermal conductivity range coupled with the parameters shown in Table 1 (E, Z, Q, R and ρ), the predicted critical temperature range for time to explosion geometry is 232.8-237.1 °C. This close match of predicted and experimental critical temperatures for the Time to Explosion test provides confidence in the thermal data gleaned from the One-Liter test and in the experimental kinetic parameters determined by the method described in Reference 7.

SECTION IV

CONCLUSIONS

The One-Liter test has been developed as a mid-scale, safety characterization test specifically for use with melt-castable energetic materials/formulations. It is a one-dimensional test carried out under relatively unconfined conditions that are similar to those encountered in melt casting operations. It provides the researcher with a materials thermal response characteristics that include the non-catastrophic self-heating and critical temperatures, the temperature at which the catastrophic, thermal decomposition event takes place and insight into the severity of this event. The experimental critical temperature from this test allows one to assess the accuracy of the value predicted by use of the F-K equation and, in so doing, corroborates (or not) the most important input parameters (E, Z and λ) used in the equation. Corroboration is important, since it is the use of the F-K equation with these important parameters incorporated that one uses to predict the critical temperature for large-scale, melt-casting operations and thusly assess the suitability of a material for scale-up.

The experimental critical temperature for octol (65/35) in one-liter geometry falls in the temperature range 152-155 °C. Using this temperature range to calculate a corresponding thermal conductivity range (1.97e-4 to 2.95e-4 cal cm^{-1} s^{-1} K^{-1}) allows one to predict a critical temperature range for the Time to Explosion test geometry of 232.8-237.1 °C. The experimental critical temperature for this latter test is 236 °C. The close match of the experimental and predicted critical temperatures associated with the Time to Explosion test lends support to the use of the One-Liter test as a mid-scale safety characterization test and to the various thermal and kinetic parameters determined for octol (65/35). While larger scale (5-gallon) melt-casting operations have been safely carried out with octol (65/35), the safety factors predicted for 30- and 100-gallon operations are only marginal (37 and 33 °C, respectively).

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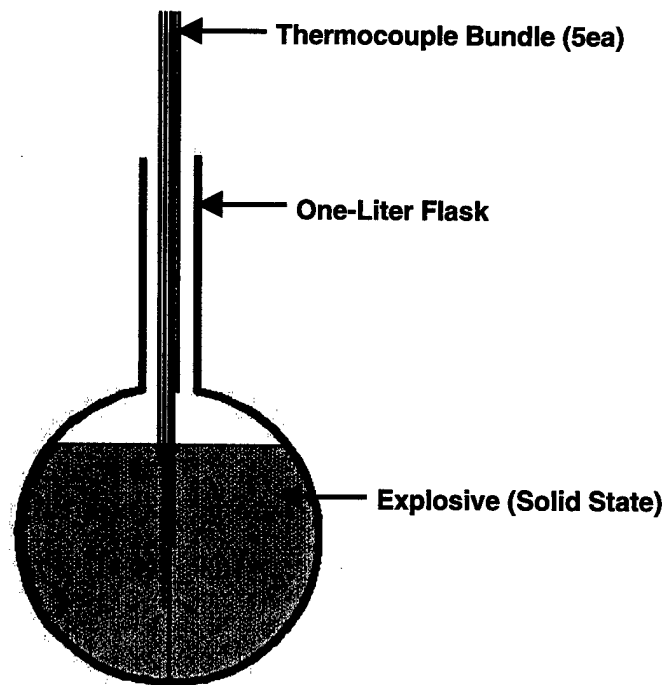
Figure 1. One-Liter Flask With Thermocouple Bundle (5) Positioned in
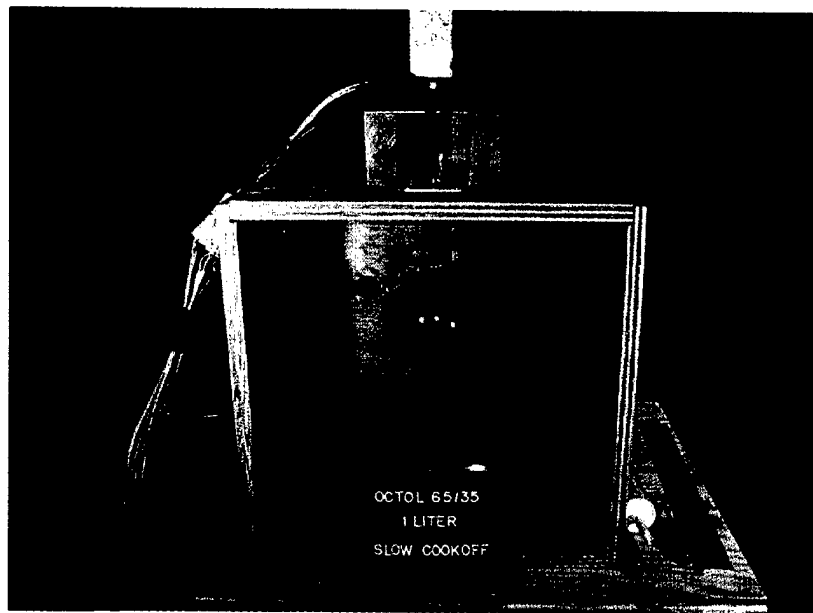Resolidified Explosive (Shaded Area)

Figure 2. One-Liter Test Setup Showing the Flask Positioned in the Plywood Oven

Sequence	Time (s)	Oven	Bottom	Mid-Bot	Center	Mid-Top	Top
0	0	23.52	19.4	19.44	21.16	21.41	20.2
1	60	23.42	19.44	19.49	21.13	21.37	20.2
2	120	23.3	19.42	19.47	21.14	21.29	20.06
3	180	23.16	19.46	19.49	21.03	21.25	19.92
4	240	26.91	19.55	19.58	21.06	21.26	20.03
5	300	35.62	19.76	19.75	21.38	21.53	20.52
6	360	43.88	20.27	20.25	22.23	22.29	21.56
7	420	51.26	20.9	20.92	23.43	23.55	22.92
8	480	57.28	21.62	21.63	24.92	25.1	24.01
9	540	62.95	22.7	22.76	26.9	27.27	26.41
10	600	68.91	23.79	23.86	29.01	29.59	28.32
11	660	73.88	24.71	24.82	30.97	31.56	29.77
12	720	78.03	25.77	25.96	33.12	33.83	31.54
13	780	82.34	26.63	26.83	34.88	35.58	32.5
14	840	85.76	27.81	28.08	37.03	37.88	34.75
15	900	89.5	29.05	29.33	39.19	40.12	37.06
16	960	92.32	30.1	30.44	41.07	42.05	38.64
17	1020	95.81	30.83	31.17	42.58	43.65	38.86
18	1080	97.88	32.46	32.82	44.96	46.16	42.79
19	1140	100.72	33.25	33.58	46.41	47.54	43.27
20	1200	102.94	34.06	34.39	47.78	48.88	43.87
21	1260	104.84	35.37	35.72	49.7	50.86	46.41
22	1320	106.99	36.24	36.52	50.97	52.16	47.13
23	1380	108.81	37.31	37.58	52.52	53.97	48.38
24	1440	110.67	38.29	38.52	53.93	55.21	49.65
25	1500	111.88	39.4	39.61	55.38	56.98	51.28
26	1560	112.81	40.1	40.19	56.36	57.73	51.24
27	1620	113.14	41.12	41.21	57.66	59.16	52.58
28	1680	114.3	42.41	42.44	59.2	60.8	54.75
29	1740	114.43	43.41	43.39	60.36	62.05	55.73
30	1800	114.44	44.45	44.35	61.46	63.01	57.21
31	1860	114.36	45.08	44.94	62.14	63.71	57.38
32	1920	113.82	45.98	45.74	62.95	64.63	58.05
33	1980	113.02	46.52	46.25	63.49	65.15	57.66
34	2040	112.52	47.28	46.92	64.04	65.57	58.57
35	2100	112.3	47.76	47.36	64.29	65.76	58.21
36	2160	110.93	48.6	48.06	64.88	65.98	59.23
37	2220	110.16	48.69	48.14	64.72	65.86	57.54
38	2280	108.77	49.79	49.17	65.65	66.91	59.36
39	2340	107.77	50.24	49.58	65.77	66.89	59.34
40	2400	106.89	50.69	49.94	65.81	66.77	59.13
41	2460	106.07	51.11	50.35	65.86	66.42	59.09
42	2520	105.27	52.02	51.18	66.44	67.07	60.94
43	2580	104.49	52.85	51.96	66.91	67.62	62.01
44	2640	103.88	52.79	51.86	66.41	66.94	60.01
45	2700	103.26	53.5	52.51	66.73	67.32	61.02
46	2760	102.64	53.59	52.59	66.46	66.96	59.82
47	2820	102.49	54.23	53.18	66.85	66.92	60.72
48	2880	102.2	54.87	53.75	67.1	67.19	61.33
49	2940	101.83	55.37	54.22	67.27	67.5	61.4
50	3000	101.83	55.97	54.79	67.51	67.59	61.92

Figure 3. Example of Acquired Temperature/Time Data
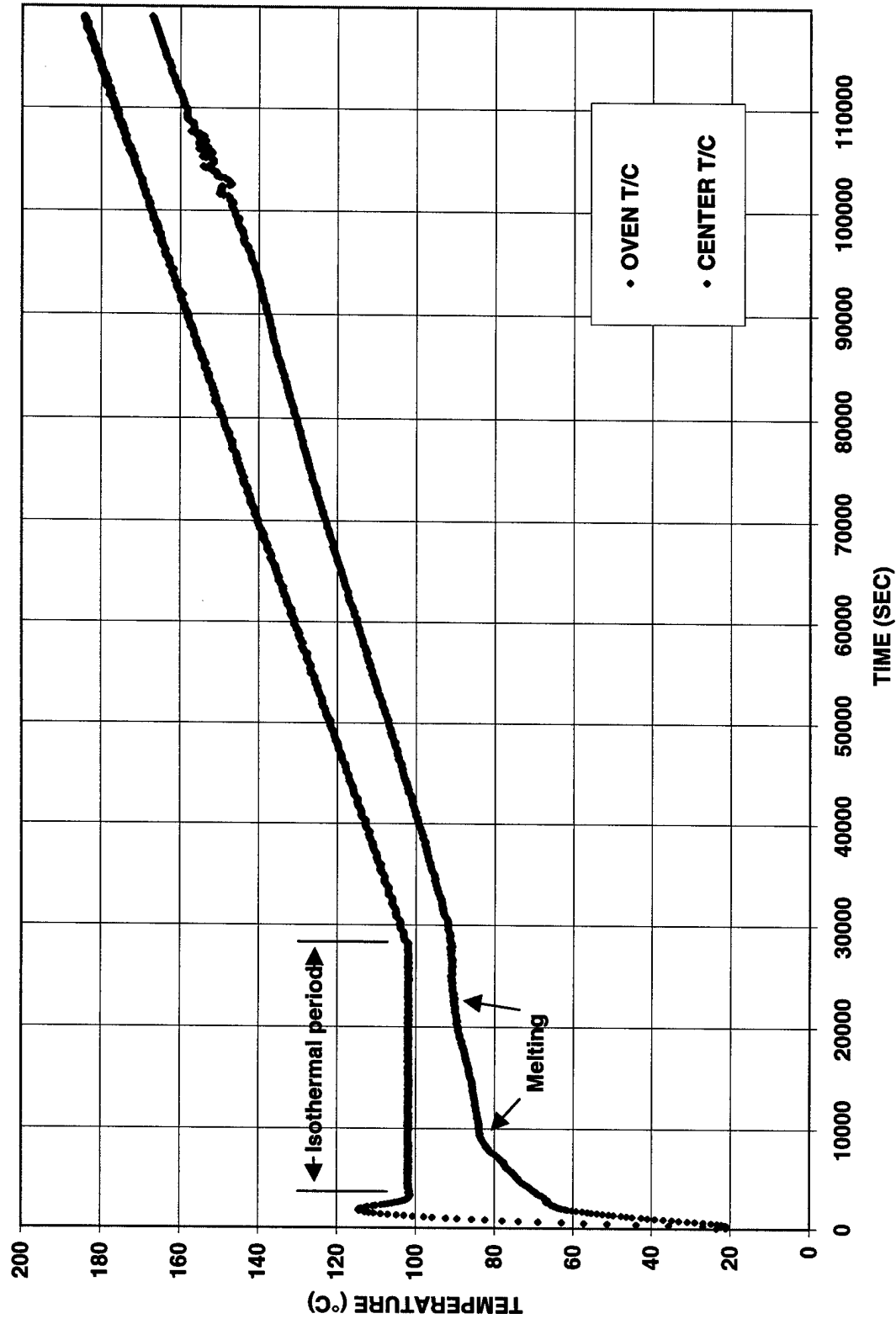
OCTOL (65/35)

Figure 4. Example of Temperature/Time Traces for the Oven and Center Thermocouples
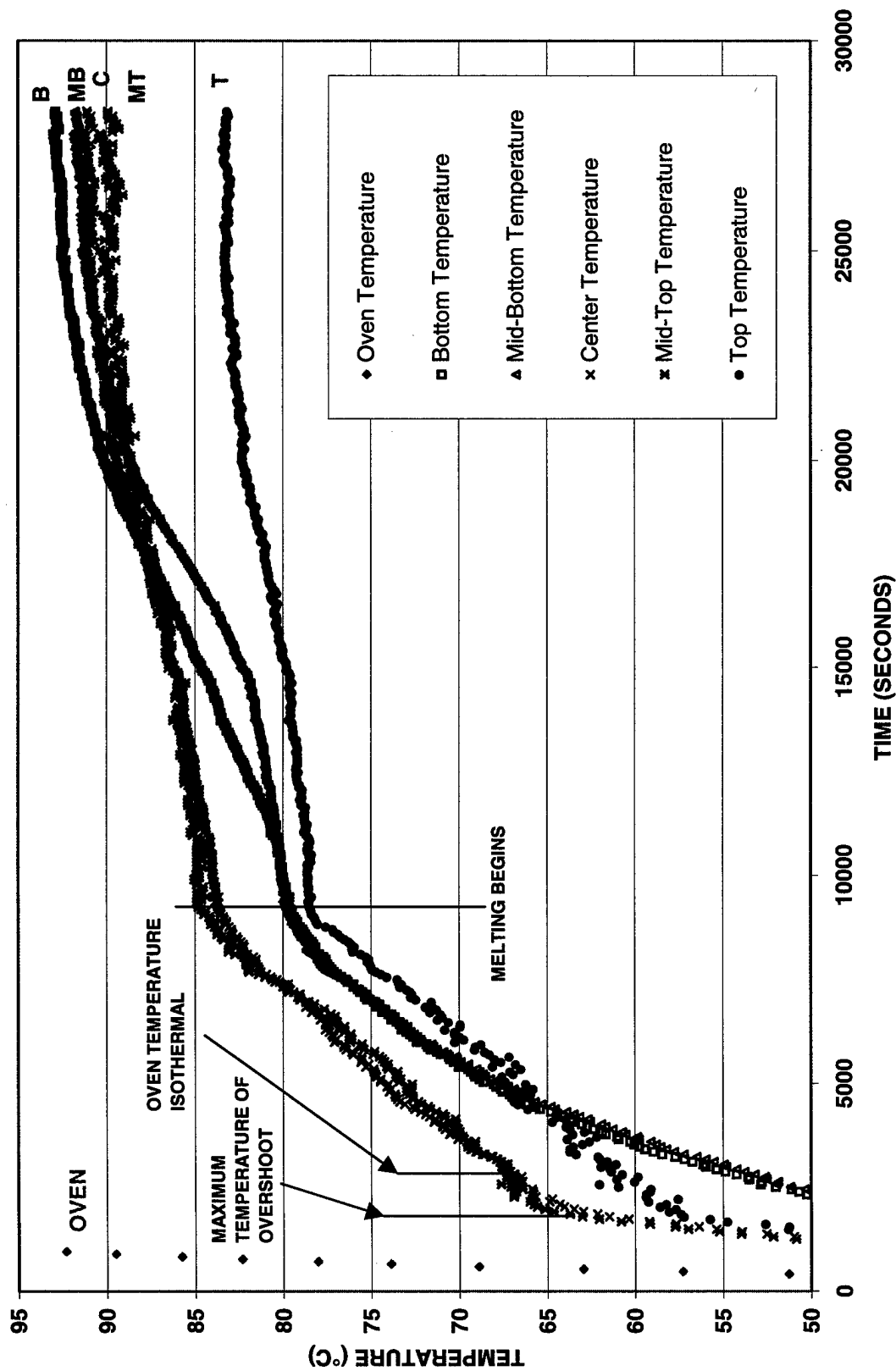
16

OCTOL (65/35)

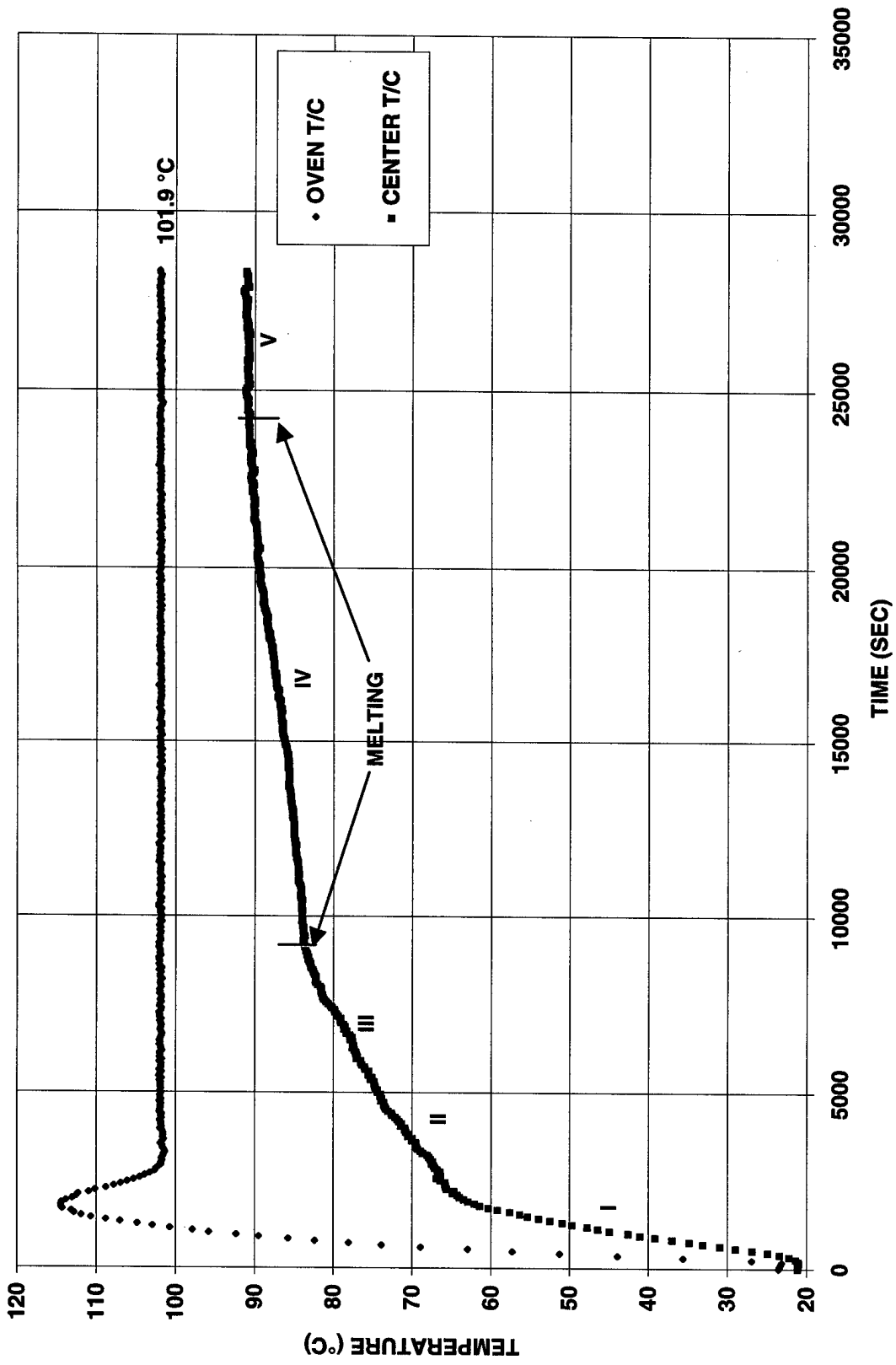Figure 5. One–Liter Test Showing the Temperature Ramp/Isothermal Periods for All Thermocouples

OCTOL (65/35)

Figure 6. Temperature Profiles for the Initial Ramp and Isothermal Conditioning Periods

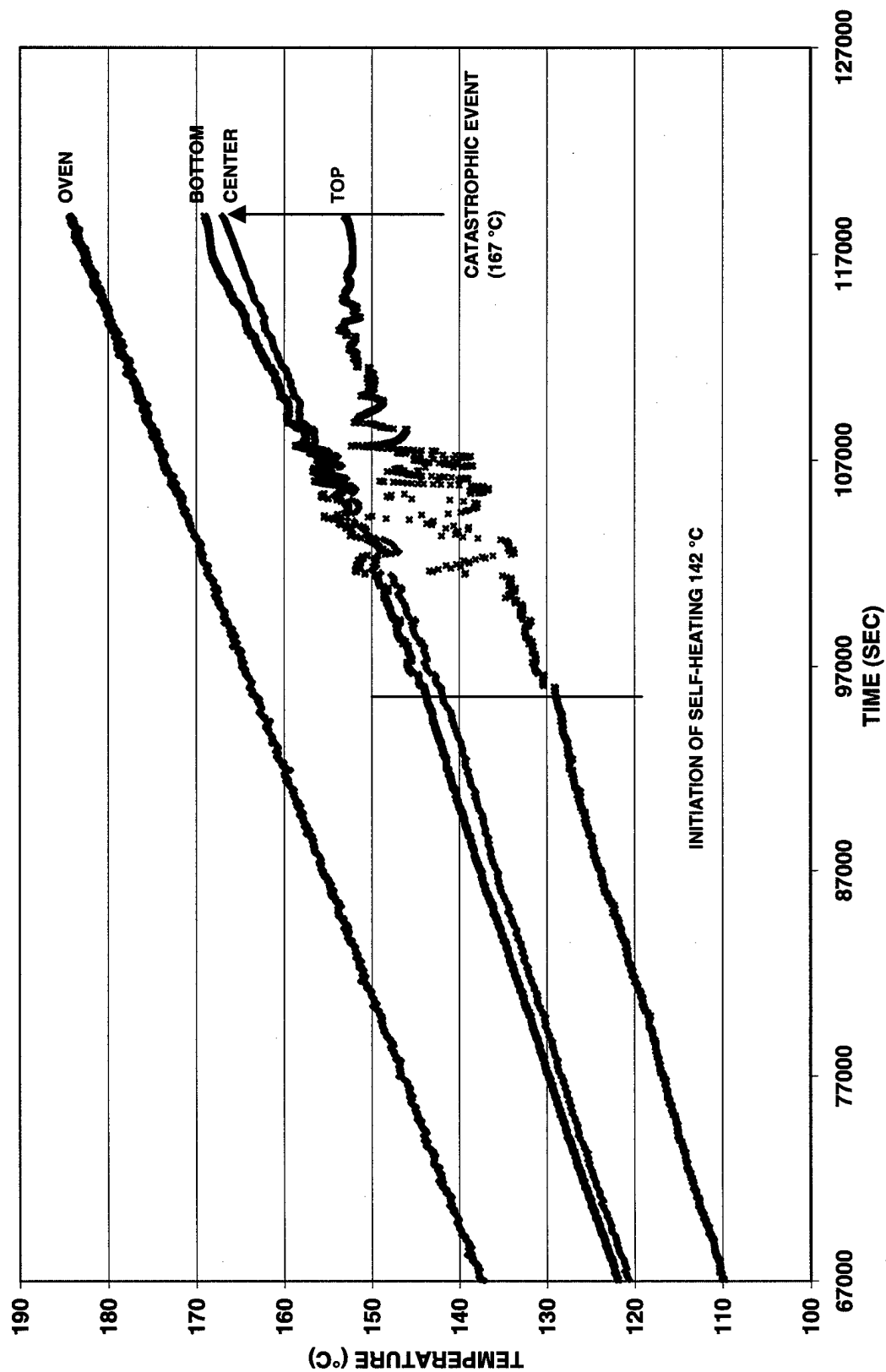Figure 7. Temperature/Time Profiles Associated with the Oven, Bottom, Top and Center Thermocouples
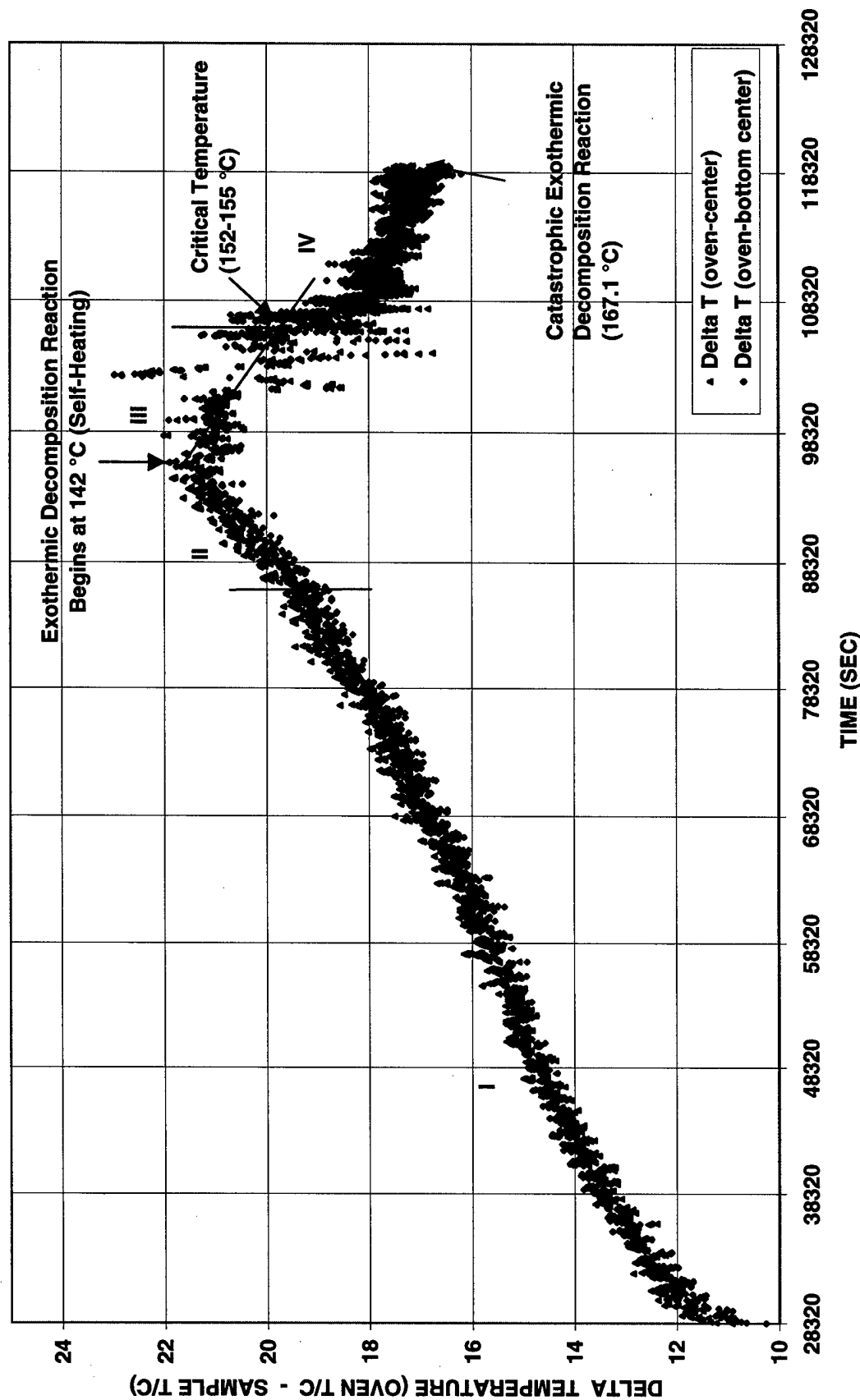
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OCTOL (65/35)

Figure 8. Delta Temperature Profile During the Final Dynamic Period
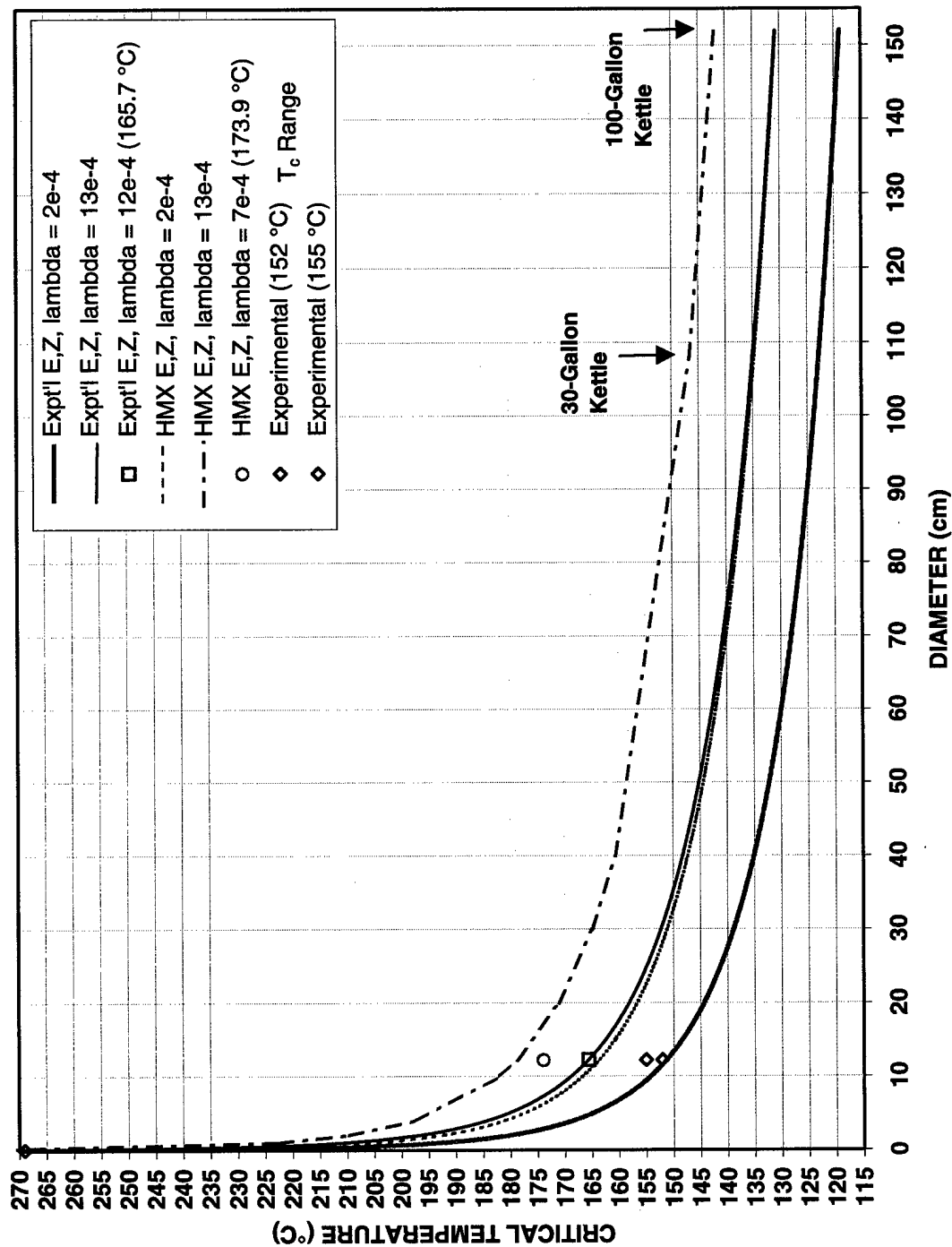
20

OCTOL (65/35)

Figure 9. Predicted and Experimental Critical Temperatures for Octol (65/35) in Spherical Geometry

21

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